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The Outlook for Some Fission Products Utilization with the Aim to Immobilize Long-Lived Radionuclides

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The prospects for development of nuclear power are intimately associated with solving the problem of safe management and removal from the biosphere of generated radioactive wastes. The most suitable material for fission products and actinides immobilization is the crystalline ceramics. By now numerous literature data are available concerning the synthesis of a large range of various materials with zirconium-based products. It worth mentioning that zirconium is only one of fission products accumulated in the fuel in large amounts.

The development of new materials intended for HLW immobilization will allow increasing of radionuclides concentration in solidified product so providing costs reduction at the stage of subsequent storage.

At the same time the idea to use for synthesis of compounds, suitable as materials for long-term storage or final disposal of radwastes some fission products occurring in spent fuel in considerable amount and capable to form insoluble substances seems to be rather attractive. In authors opinion in the nearest future one can expect the occurrence of publications proposing the techniques allowing the use of ‘reactor’s zirconium, molybdenum or, perhaps, technetium as well, with the aim of preparing materials suitable for long-lived radionuclides storage or final disposal.

The other element, which is generated in the reactor and worth mentioning, is palladium. The prospects for using palladium are defined not only by its higher generation in the reactor, but by a number of its chemical properties as well.

It is evident that the use of natural palladium with the purpose of radionuclides immobilization is impossible due to its high cost and deficiency). In author's opinion such materials could be used as targets for long-lived radionuclides transmutation as well.

The object of present work was the study on methods that could allow to use "reactor" palladium with the aim of long-lived radionuclides such as I-129 and TUE immobilization.

In the paper the results of experiments on synthesis of matrices with TUE oxides and PdI_2 on palladium base are presented.

INTRODUCTION

The major way for increasing the safety of radwastes management is the development of advanced technologies, which would enable to reprocess the whole volume of high-level waste (HLW) and to obtain the solid products offering high chemical stability which would be suitable for long-term monitored storage and/or final disposal.

The use of the partitioning techniques in combination with synthesis of mineral-like materials provides highest possible safety during subsequent storage and final disposal all of kinds of radioactive wastes [1-4]. All possible changes in process structure and in the usage of new reagents and techniques must fulfil the task of subsequent separation of individual radionuclides (or their fractions) and their solidification in the forms, which satisfy the requirements for their final removal from biosphere. Solving this problem would be the goal of all researches for the nearest decades.

At the same time the idea to use for synthesis of compounds, suitable as materials for long-term storage or final disposal of radwaste some fission products occurring in spent fuel in considerable amount and capable to form insoluble substances seems to be rather attractive.

Along with zirconium, the other elements, such as molybdenum, platinum group metals are found in the fuel and could be used for synthesis of various compounds of short-lived (Cs, Sr) or long-lived (TUE) radionuclides.

One of such elements is palladium; it's accumulation in spent PWR fuel, coming to 1.0 - 1.8 kg per ton. (In FBR reactors where fuel burnup is higher, "artificial" palladium generation is more intense and palladium content in FBR's fuel is about several kilograms per ton [5-6].)

The first ideas related to prospects of platinum group metals recovery from spent fuel and their utilization appeared as early as 50 years ago [7-9]. However, several decades later it was quite obvious that the occurrence of palladium radionuclide ^{107}Pd (half-life $6.5 \cdot 10^5$ years) in "reactor" palladium would exclude the possibility of its use in common industry.

Various techniques can be used for palladium recovery from spent fuel [10-14]. Till recently the raffinate of Purex-process was considered as the only product suitable for palladium recovery and the main part of the investigations was directed to the development of the techniques for palladium recovery precisely from these products. At the same time, the technology of HLW partitioning which is being developed in recent years, provides a precondition for the use of an alternative source for palladium recovery: the solutions resulted from this operation are lesser active and lesser complicated as compared with the raffinates of the I extraction cycle, comprising the total sum of fission products. And from resulted solution palladium black can be obtain by means of precipitating technique [14].

One could suppose that the inclusion of the radioactive elements oxides or slightly soluble iodine compounds in the matrix of metal palladium would allow to produce materials with low leaching rate and suitable for a safe long-term storage or final disposal. In author's opinion such materials could be used as targets for long-lived radionuclides transmutation as well. It seems advisable to examine in future the opportunity of technetium usage for solving problems of HLW immobilization. (In author's opinion it's of interest to use technetium as material to obtain protective covering on granulated wastes or to encapsulate solidified HLW.)

The object of this work was to carry out a study on searches of engineering decisions, which would allow using of 'reactor' palladium for immobilization of such long-lived radionuclides as I-129 and transplutonium elements (TPE). Are presented in this paper the results of experiments on Pd-based metalloceramic compositions synthesis, these compositions including metal oxides or palladium iodide, and the results of electron microprobe analysis and lixiviation rates for Eu and I are given.

EXPERIMENTAL RESULTS AND DISCUSSION

The synthesis of Pd-containing matrix compositions intended for TPE immobilization.

The most special palladium feature which differs this element from the other platinoides is its capacity to be dissolved into nitric acid. Therefore, if in the future the materials on Pd-base would be used for TUE immobilization and long-term storage, a simple dissolution of these mateials in nitric acid followed by extraction recovery of target elements (americium , curium…) would be sufficient in case of TUE recovery need. It was supposed that the incorporation of oxides of radioactive elements recovered from the wastes of various levels into the matrix of metal palladium would allow to decrease considerably their leachibility and to organize a long-term safe storage of these radionuclides. (It is obvious in the case if we include TUE into mineral-like matrices we lose opportunity to recover them forever.) Subsequently these materials could be used for the transmutation of TP element radionuclides.

In the first stage the experiments were carried out on palladium powder pressing. The goal of these studies was to find out the effect of binding agent should be added on mechanical strength of pressed pellets. (The solution of paraffin in heptane was used as a binder. Binder's mass was 0.2-3.0% of metal palladium powder mass.)

The mechanical strength of the specimens so obtained was further analyzed. The results of these experiments allow making the following conclusions.

The comparison of the specimens being produced when using various amounts of binder did not reveal any significant differences.

Among all tested techniques for powder compacting the heat-treatment at 1000°C only has led to a plastic deformation of the specimen. In the other cases the pellets underwent a brittle fracture through disintegration into powder. The appearance of the pellet after mechanical test is shown in Fig. 1.

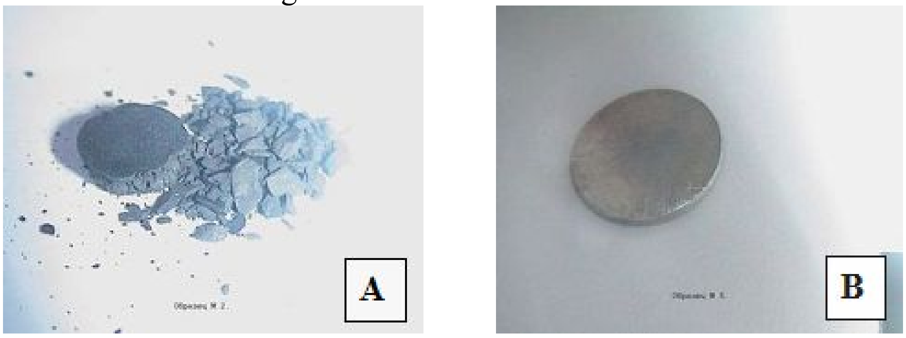
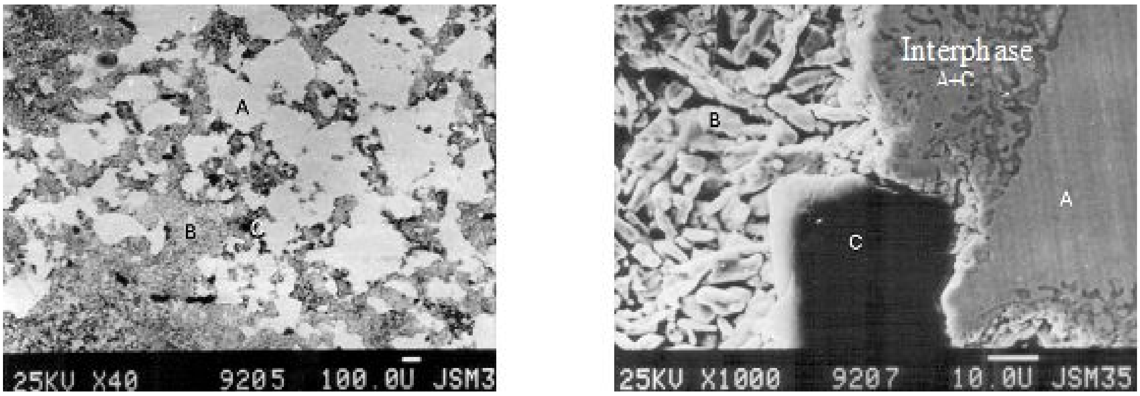


Fig. 1. The appearance of the pellets being pressed from metal palladium powder and further tested for strength. Pellet production technique: (A) - pressing with the adding of binding agent (1% solution of paraffin in heptane); (B) - pressing with no binder, followed by sintering at 1000°C.

In the next stage of our studies the experiments on Pd- and Eu_2O_3-based product synthesis were conducted.

Weighted portion of working mixture (3-4 g, metal palladium and europium oxide) was poured into press-mold. (The mold was inserted inside the inductor. The chamber was evacuated by means of fore pump and further filled with argon.) The mixture of metal palladium and europium oxide was subjected to cold pressing by the pressure of 300 kgf/cm^2. After pressure release the mould induction heating started. The temperature interval has varied from 300°C till 1000°C Pressing length was as large as 1 hour.



302-1, (Reflected electrons): A-Pd: S/S_0=0.5, B-Eu_2O_3, C-X phase: S/S_0=0.07, L_C=40x80 μ, S/S_0 - relative area of phase

302-1, (Secondary electrons): C-X phase - L_B=3x7 μ (Size of Eu_2O_3 crystallites), Interphase: solid solution (Pd-Eu_2O_3)

Fig.2. The results of electron-microscopic analysis for pellets with Eu_2O_3. Specimen #302-1. (50 mass.% Pd + 50% mass.% Eu_2O_3)

At micrographs obtained three phase (A, B, C) were clearly seen which are non-uniformly distributed throughout the pellet surface. A comprises mainly metal palladium whereas the phase B is constituted of europium oxide. The phase C is the most likely to be a solid solution of Pd- Eu_2O_3 and an X-ray amorphous substance (the most likely – carbon of graphite press-mold). (Europium oxide and palladium contents were consistent with composition of initial mixture being pressed.)

The leach rate was determined for all specimens. These tests were conducted in distillated water, room temperature. In all cases the europium concentration was at the limit level of this technique sensitivity or is below it. Consequently these data must be considered as evaluating ones. However, calculated values of leaching rates are at the level of 10^{-5} – 10^{-6} kg/m^2·day.

Literature data show that the leaching rates of uranium and plutonium from common matrices based on glass and ceramics of various types are at the level of 10^{-3} – 10^{-5} kg/cm^2·day and those of REE and TPE are at the level of 10^{-5} kg/m^2·day [4, 15-19]. (According to the requirements of Russian standard "GOST # 50926-96" the velocity of actinides (Pu) leaching from the matrices used for HLW long-term storage should be at the level of 10^{-5} kg/m^2·day.)

With the purpose of studying the possibility of matrix composition ($PdxEo_2O_3$) chemical stability strengthening the pressed pellets were subjected to a chemical "palladiaizing". Two specimens of $PdxEu_2O_3$ with Eu_2O_3 content of 50 and 70 mass.% were treated. The specimens were preliminary degreased with alcohol and then dried up to the termination of any specimen weight variation. It was found that the pellet containing 70 mass.% Eu_2O_3 had a noticeable porosity (an air escape from the pores was observed during specimen treatment by alcohol).

Traditional procedure generally used for was applied for palladium coating production. Operation conditions were as follows: the electrolyte composition (g/L): palladium chloride – 3÷5, 25% aqueous ammonia – 15÷30, sodium hyposulfite – 10÷30, sodium thiosulfate – 0,025÷0,035; temperature, °C – 40-60.

The pellets being noticeably porous, the determination of palladium layer depth basing upon the mass of deposed metal didn't provide sufficient precision. Consequently, palladium plating was conducted for 2 hours at 50°C, so as to obtain palladium layer about 10 micrometers in depth (calculated for overall geometric surface of the pellet). These operational conditions were established after preliminary experiments on palladium deposition on platinum foil. After specimens rinsing with water and drying the calculated depth values for pellets with 50% and 70% Eu_2O_3 were 6,3 and 5,9 μ respectively. Pellet's appearance remained essentially unchanged after this treatment. These specimen structure and chemical stability were being studied. The results of encapsulated specimens analysis reveal that palladium coating had blocked (D_{block} ~ 200 μ) porous structure (Dp~3 μ The range of porosity) and was inhomogeneous in composition due to local variations of coating depth. Most likely coating of metal palladium had no through cracks but its depth varied within wide limits (from 0,1 to 1,5 μ).

Encapsulated pellets were subjected to leaching using static method. Experimental procedure differed from that described above in that the sampling was carried out without renewal of the solution over pellets. The pellets without coating were tested in the same manner simultaneously with encapsulated ones (Table I).

Table I. The leach rate of europium from encapsulated Pd-base metalloceramic compositions.

Pallet content	t, days	Eu in solution, mg/l	R, kg/m^2·day
50% Pd +50% Eu_2O_3	202	110	$1.4 \cdot 10^{-4}$
(50% Pd +50% Eu_2O_3)**Pd**	202	40	$5.1 \cdot 10^{-5}$
30% Pd +70% Eu_2O_3	111	13	$2.3 \cdot 10^{-5}$
(30% Pd +70% Eu_2O_3)**Pd**	111	0,25	$4.6 \cdot 10^{-7}$

The coating of the pellets with metal palladium (even if porous) results also in reducing of europium leaching rate that may decrease to $\sim 5\cdot 10^{-7}$ kg/m$^2\cdot$day in case of encapsulated pellet containing 70 % europium oxide.

Our experimental data show that chemical stability of the compositions under examination is as good as that of common matrices. Therefore, the use of these materials for radionuclides long-term storage and transmutation seems to be quite justified, particularly, when it is considered that radionuclides incorporated into palladium compositions could, if required, be extracted there from by means of common techniques.

Experimental studies on iodine–129 immobilization with the use of 'reactor' palladium.

Presently the problem of radioiodine management receives higher attention in connection with increasing stringency of requirements for environmental protection.

The requirements to materials intended for preparing of targets for ^{129}I transmutation are essentially the similar to those applied for TPE transmutation (chemical and mechanical stability to heating and irradiation, compatibility with cladding and heat carrier). Considering that rather low ^{129}I transmutation degree is achieved upon a single transmutation cycle (several percent), a complete ^{129}I transmutation would need several cycles, that is to say that iodine should be recovered from irradiated target and further directed again to transmutation.

The choice of materials suitable for iodine transmutation is performed among iodides, iodates and oxiiodides [20-22]. The literature cites slightly soluble palladium iodide, which is quite suitable for long-term storage and/or final disposal of iodine radionuclides [23].

At the first stage of the work the conditions of palladium iodide deposit manufacture from nitrate-acid solutions have been studied. A precipitate of PdI_2 was obtained by mixing of solutions of KI and Pd^2 in nitric acid. (HNO_3 concentration – 2 mole/l.) The value of $Pd^{2+}/2I^-$ ratio varied during the experiments from 5 to 50% of palladium excess relative to stoichiometric of reaction. However the yield of palladium iodide precipitates was almost the same in the majority of these experiments (98,1 – 98,6%). Electron-microscopic photos of the specimens are given in Fig. 3. Within the limits of experimental error I/Pd ratio value for all specimens agrees with stoichiometric ratio for PdI_2. (The other elements have not been detected, whereas the occurrence of non-analyzable elements in amounts below 2-6% is possible.)

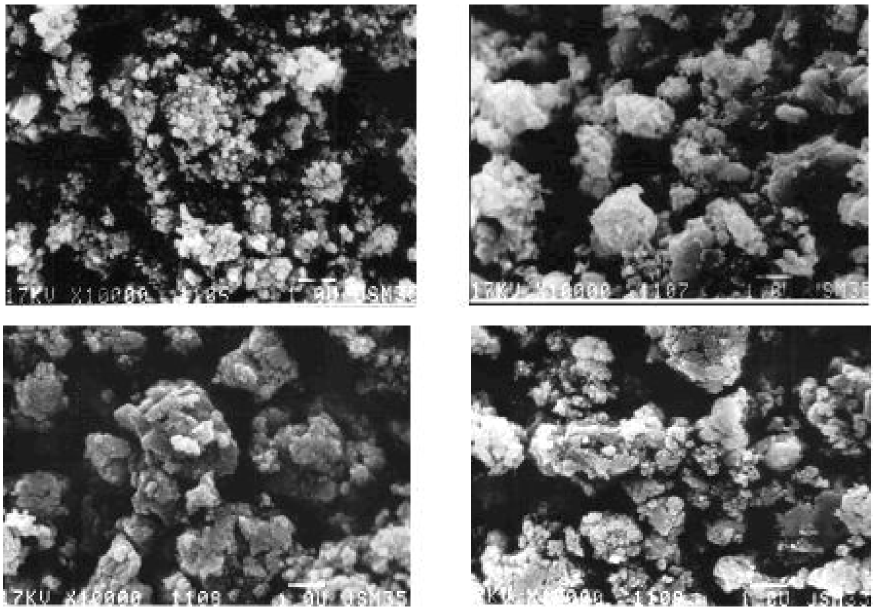


Fig. 3. Electron-microscopic photos of PdI_2 specimens.

Substantial characteristic of iodine compounds intended for preparing of targets for ^{129}I transmutation is chemical and heating stability.

Another objective of conducted researches was palladium iodide (PdI_2) solubility determination with the use of radiotracer I^{125} (half-life period 60 days). ^{125}I was added as the "marker" during PdI_2 specimen's synthesis. The value of PdI_2 solubility was found to be as low as 2,5 mg/l.

At the next stage of the work we prepared the specimens containing 50% palladium iodide being incorporated into metal palladium matrix.

With the purpose of studying the possibility of matrix composition ($PdxPdI_2$) chemical stability strengthening the pressed pellets were subjected to a chemical "palladiaizing". The specimen $PdxPdI_2$ with PdI_2 content of 50 mass.% were treated. Pellet's appearance remained essentially unchanged after this treatment. These specimen structure and chemical stability were being studied. Encapsulated pellets were subjected to leaching using static method. (Experimental procedure differed from that described above in that the sampling was carried out without renewal of the solution over pellets.)

The pallet with 50% Pd + 50% PdI_2 (mass= 2.814 г, Ø = 10 mm, h = 7.67 mm) was using in the first experiment. It was put into teflon glass and fill up by distilled water. The time of experiment was 210 days without solution changing. At regular intervals samples have been taken and the content of iodine and palladium were determined. (Detection limit of iodine is 0.001 mg/l, Palladium – 0.0002 mg/l).

The results of this experiment are given in Table II.

Table II. The results of this experiment on iodine leaching.

t, days	I, mg/l	Pd, mg/l	R, kg/m^2 day
26	1.4	83.3	$8.98 \cdot 10^{-4}$
78	0.8	154	$1.67 \cdot 10^{-4}$
210	0.56	209	$4.22 \cdot 10^{-5}$

According the data shown in this table the content of palladium is increased in the course of experiment something in time 2,5 times and at the same time content of iodine is decreased. This fact could be explained in the following way. In the presence of water $[PdI_4]^{-2}$ is got out in solution but for all that the part of iodine is retained in matrix of pallet with metallic palladium.

In the second set of experiments the pallets were manufactured with using cool pressing technique. The content of palladium iodide was 30, 50 and 70 masses % accordingly.

For the purpose chemical stability comparison the pallets on the base of copper and palladium iodide were manufactured.

Presented data point out to the large chemical stability of compositions on metallic palladium base. Initial leaching-out rates of iodine come to level $2 \cdot 10^{-4}$ - $8 \cdot 10^{-5}$ kg/m^2 day.

During further aging, leaching-out rates go down and come to $8 \cdot 10^{-7}$ - $9 \cdot 10^{-6}$ kg/m^2 day depending on palladium iodide content. In the case of palladium based composition lower leaching-out rates are observed in comparison with copper based compositions.

The results of the subsequent investigation on kinetics of iodine leaching from palladium pellets are presented in Table III.

The experiments on the determination of palladium and iodine leaching rate from the pellets being subjected to the chemical "palladiumization" (Tabl. IV) have been also carried out.

The presence of protective superficial palladium coating gives rise to considerable increase of chemical stability. Palladium content in the leach rate decreases by 6 orders of magnitude and that of iodine — by 3 – 5 times. (It should be noted iodine lixiviation rate from phosphate glasses is of the same order [24].

Table III. Comparison of chemical stability of compositions on the base of palladium without coating and with metal copper coating after the contact with distilled water.

Speci-mens #	Composi-tion	Characteristics of pellet				Time of aging, days		Content of elements in solution, mg/l			R, kg/m^2*day
		M, g	H, мм	D, мм	S, sm^2	Δ t, days	T total time days	Pd	Cu	I	
403	**50% Pd + 50% PdI_2**	1.50	3.4	10.0	3.70	13	13	0,029	-	≤0,003	8.9E-08
						8	21	0,004	-	≤0,003	1.4E-07
						26	47	0,009	-	≤0,003	4.4E-08
						51	98	0,041	-	≤0,003	2.3E-08
404	**(50% Pd + 50% PdI_2)Cu**					8	21	≤0.001	0.38	≤0.003	2.2E-07
						26	47	≤0.001	0.64	≤0.003	6.6E-08
						51	98	≤0.001	1.36	≤0.003	3.4E-08
405	**30% Pd + 70% PdI_2**	1.49	3.5	10.0	3.77	13	13	0.041	-	≤0.003	6.3E-08
						8	21	0.017	-	≤0.003	1.0E-07
						26	47	≤0.001	-	≤0.003	3.1E-08
						51	98	≤0.001	-	≤0.003	1.6E-08
406	**(30% Pd + 70% PdI_2)Cu**	5.48	6	15	9.18	13	13	≤0.001	2.97	≤0.003	9.5E-08
						8	21	0.004	0.62	≤0.003	1.5E-07
						26	47	0.022	0.064	≤0.003	4.7E-08
						51	98	0.028	0.036	≤0.003	2.4E-08

Table IV. The results of the experiment on the determination of iodine leaching rate from the pellets with palladium coating.

The pallet content	t, days	I, mg/l	Pd, mg/l	R, k/m^2·day
50 % Pd + 50 % PdI_2	26	1.4	83.3	$8.98\cdot10^{-4}$
	78	0.8	154	$1.67\cdot10^{-4}$
	210	0.56	209	$4.22\cdot10^{-5}$
(50 % Pd + 50 % PdI_2)Pd	26	0.45	0.004	$2.89\cdot10^{-4}$
	78	0.59	0.003	$1.23\cdot10^{-4}$
	210	0.12	0.008	$9.05\cdot10^{-6}$

Thus, the results of the experiments have dramatically demonstrated the possibility of producing the matrix metal-ceramic compositions based on metal palladium containing iodine compound inclusions.

At the final stage of the work we had executed the calculation of neutron physical characteristic of Pd-based matrix (from the point of view the behavior under irradiation) has been executed. There is only one radioactive nuclide of palladium in spent nuclear fuel: Pd-107 which has a half-life of $6{,}5\cdot10^5$ years and decays with radiation of soft beta rays of 35 keV. One of possible "reactor" palladium usage that waives an radioactivity objection connected with its radioactivity is palladium usage as a basis for making containers and targets for long-term storage long-lived radioactive materials and irradiation in reactor (transmutation). (Melting temperature (1552° C), mechanical strength and also chemical inertness permit to use palladium as target's material.)

While estimation Pd container's neutron physical characteristic from the point of view the opportunity of using for inter reactor it should be noted the following:

Container not must insert fair quantities of distortion in neutron flow in reactor channel.

The data for palladium isotopic composition in spent fuel and neutron-absorption cross-sections are presented in Table V below. (Data of JENDL 3.3. library have been used.)

Neutron-absorption cross-sections of Pd are notably higher in comparison with constructional materials of reactor active zone (zirconium iron or nickel). However container's influence isn't catastrophically and neutron flow reduction in immediate vicinity will average about 15 – 20%.

Table V. Isotopic composition of palladium in spent fuel and neutron-absorption cross-sections.

Nuclide mass	Half-life	Content in spent fuel after 30 years (%, weight)	Neutron-absorption cross-sections, barns		
		30 years	Thermal neutrons	Intermediate neutrons	Fast neutrons
^{102}Pd	Stable	0.0	2.97	19.5	0.11
^{104}Pd	Stable	15,8	0.46	21.9	0.077
^{105}Pd	Stable	27,5	17.9	96.7	0.12
^{106}Pd	Stable	26,4	0.27	9.3	0.073
^{107}Pd	$6{,}5 \cdot 10^5$ years	15,9	1.8	112.2	0.122
^{108}Pd	Stable	10,9	7.6	252.1	0.054
^{110}Pd	Stable	3,6	0.2	2.81	0.028

While palladium irradiation some long-lived nuclides (silver, gadolinium) could be accumulated. According rough estimate during one year of irradiation it could be accumulated about 0.1% atoms of silver and gadolinium that give a few curies per gram of palladium.

Accordingly neutron physical characteristic of Pd are severely limited but inestimable advantage connected with simple reprocessing of Pd-based targets after irradiation (TPE transmutation) give grounds to consider this material as very promising.

CONCLUSION

Considering the future of spent fuel reprocessing it is necessary to underline that the main stream in its development should be aimed at waste treatment. All possible changes in process structure and in the usage of new reagents and techniques must fulfill the task of subsequent separation of individual radionuclides and their solidification in the forms, which satisfy the requirements of their final removal from biosphere. Solving this problem would be the goal of all researches for the nearest decades.

These preliminary experimental results allow conclusion that the use one of fission product - 'reactor' palladium as a material for I-129 and TPE immobilization seems to be quite justified. (It is evident that the use of natural palladium is impossible due to its high cost and deficiency). At the same time the use of palladium in radioactive waste reprocessing flowsheet (where the presence of radioactive ^{107}Pd nuclide is of no importance), could play a main role in the creation of the demand on this metal.

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